

# PATENT SPECIFICATION

NO DRAWINGS

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## COMPLETE SPECIFICATION

### A process for the production of Dibasic Organic Di-Per Carboxylic Acids

5 We, DEUTSCHE GOLD- UND SILBER-SCHEIDANSTALT VORMALS ROESSLER, of 9, Weissfrauenstrasse, Frankfurt (Main) 1, Germany, a body corporate organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to a process for the production of dibasic organic di-per acids.

15 British Patent 791,875 describes a process for reacting aliphatic monocarboxylic acids with concentrated aqueous hydrogen peroxide in the presence of boron fluoride or boron fluoride monohydrate to form the corresponding per fatty acids.

20 In the further development of this process, it has now been found to be possible to produce dibasic organic di-per carboxylic acids in an analogous manner. The procedure according to the invention is that an organic dicarboxylic acid is reacted with concentrated 25 hydrogen peroxide in the presence of boron trifluoride or boron trifluoride monohydrate. The exothermic reaction takes place rapidly

The carboxylic acids can also be replaced by their anhydrides.

30 It has further been found that it is expedient to use at least two mols, and advantageously more than 2.5 mols, of hydrogen peroxide to 1 mol of the acid. It is advantageous in this case to work with highly concentrated aqueous solutions of hydrogen peroxide, these 35 solutions containing at least 90% by weight of  $H_2O_2$ .

40 It is further recommended to employ more than 1 mol and advantageously 1.2 to 2.5 mols of boron trifluoride to 1 mol of acid.

It is preferred for the better homogenisation of the reaction mixture, to add concentrated sulphuric acid.

45 The di-per acids prepared by the process according to the invention can be used for oxidation reactions, epoxidation reactions, bleaching and disinfecting purposes.

The invention is further illustrated by the following examples.

#### EXAMPLE 1.

50 1.2 parts by weight of succinic acid are dissolved in 1 part by volume of boron fluoride monohydrate and 1 part by volume of concentrated sulphuric acid is added. While 55 maintaining a temperature of 30 to 40° C., the mixture has slowly added thereto 0.5 part by volume of an aqueous solution of hydrogen peroxide, which contains 93 parts by weight of  $H_2O_2$  in 100 parts by weight of liquid. After 15 minutes, the substance 60 is poured on to ice and the precipitated acid is sharply suction-filtered. 1.1 parts by weight of white crystals are obtained, which explode violently on heating and contain, by analysis, 97.35% by weight of di-per-succinic acid and 65 0.25% by weight of free hydrogen peroxide.

#### EXAMPLE 2.

70 1.35 parts by weight of glutaric acid are reacted, according to Example 1 with 1 part by volume of boron trifluoride monohydrate, 1 part by volume of concentrated sulphuric acid and 0.5 part by volume of 93% by weight hydrogen peroxide. 1.2 parts by weight of di-per-glutaric acid are obtained in the form of white crystals, which are shown 75 by analysis to contain 92.8% by weight of di-per-glutaric acid and 0.3% by weight of free hydrogen peroxide.

#### EXAMPLE 3.

80 7.3 parts by weight of adipic acid are dissolved in 8.6 parts by weight of boron trifluoride monohydrate and 0.3 part by volume of an aqueous hydrogen peroxide solution containing 135 parts by weight of  $H_2O_2$  in

100 parts by volume is added thereto. The mixture is left standing for 45 minutes at room temperature, the crystal magna which has formed is then diluted with some iced water, allowed to settle and then washed with a little iced water. 6.5 parts by weight of di-per-adipic acid are obtained in the form of white crystals, which melt with decomposition at 106° C. and explode in a flame. After analysis, the crystals contain 90.0% of di-per-adipic acid and 0.3% of free hydrogen peroxide.

#### EXAMPLE 4.

1.0 part by weight of sebacic acid are reacted according to Example 3 with 1 part by volume of boron trifluoride monohydrate and 0.5 part by volume of 93% by weight hydrogen peroxide. 0.9 part by weight of di-per-sebacic acid is obtained in the form of white crystals, which contain 95.4% by weight of the di-per-sebacic acid and 0.3% by weight of free hydrogen peroxide.

#### WHAT WE CLAIM IS:—

1. A process for the production of dibasic organic di-per-carboxylic-acids, wherein an organic dicarboxylic acid is reacted with concentrated hydrogen peroxide in the presence of boron trifluoride or boron trifluoride monohydrate.

2. A process as claimed in Claim 1, wherein the anhydride of the dibasic acid is used.

3. A process as claimed in Claim 1 or 2, wherein at least 2 mols and advantageously more than 2.5 mols of hydrogen peroxide are used to 1 mol of acid.

4. A process as claimed in any of Claims 1 to 3, wherein the solution of hydrogen peroxide used is at least a 90% solution.

5. A process as claimed in any of Claims 1 to 4, wherein more than 1 mol and advantageously 1.2 to 2.5 mols of boron trifluoride are used to 1 mol of acid.

6. A process as claimed in any of Claims 1 to 5, when carried out in the presence of concentrated sulphuric acid.

7. A process for the production of dibasic organic di-per carboxylic acids substantially as described with reference to any of the examples.

8. Dibasic organic di-per carboxylic acids whenever produced by the process claimed in any of the preceding claims.

#### ELKINGTON & FIFE,

Consulting Chemists and Chartered  
Patent Agents,  
Bank Chambers, 329, High Holborn,  
London, W.C.1,  
Agents for the Applicants.